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PTO/SB/13 (11-96)
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REQUEST FOR FILING A PATENT APPLICATION UNDER 37 CFR 1.60

DOCKET NUMBER	ANTICIPATED CLASSIFICATION OF THIS APPLICATION		PRIOR APPLICATION EXAMINER	ART UNIT
	CLASS	SUBCLASS		
861-001-9-1			Examiner Parker	

Address to:
Assistant Commissioner for Patents
Washington, D.C. 20231

This is a request for filing a ☐ continuation ☒ divisional application under 37 CFR 1.60, of pending prior Application Number 08/813055, filed on 03/07/1997 entitled CONTINUOUS SOLID STATE WEB COATING PROCESS AND WEBS PRODUCED THEREBY

1. Enclosed is a copy of the latest inventor-signed prior application, including a copy of the oath or declaration showing the original signature or an indication it was signed. I hereby verify that the papers are a true copy of the latest signed prior application number 08/813055, and further that all statements made herein of my own knowledge are true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

CLAIMS	(1) FOR	(2) NUMBER FILED	(3) NUMBER EXTRA	(4) RATE	(5) CALCULATIONS
	TOTAL CLAIMS (37 CFR 1 16(c))	10 - 20 =	0	x $ =	$ 0.00
	INDEPENDENT CLAIMS (37 CFR 1 16(b))	5 - 3 =	2	x $ 80 =	160.00
	MULTIPLE DEPENDENT CLAIMS (if applicable) (37 CFR 1 16(d))			+ $ =	
			BASIC FEE (37 CFR 1 16(a))		+ 770.00
			Total of above Calculations =		930.00
	Reduction by 50% for filing by small entity (Note 37 CFR 1.9, 1.27, 1.28).				
				TOTAL =	$ 930.00

2. ☐ A verified statement to establish small entity status under 37 CFR 1.9 and 1.27
   ☐ is enclosed.
   ☐ was filed in prior application number ____ / _____ and such status is still proper and desired (37 CFR 1.28(a)).
3. ☒ The Commissioner is hereby authorized to charge any fees which may be required under 37 CFR 1.16 and 1.17, or credit any overpayment to Deposit Account No. 23-0442. A duplicate copy of this sheet is enclosed.
4. ☒ A check in the amount of $ 930.00 is enclosed.
5. ☒ Cancel in this application original claims 1-15 of the prior application before calculating the filing fee. (At least one original independent claim must be retained for filing purposes.)
6. ☒ The inventor(s) of the invention being claimed in this application is (are): Evan E. Koslow; Richard D. Kendrick; and Gordon Spilkin
7. ☐ This application is being filed by less than all the inventors named in the prior application. In accordance with 37 CFR 1.60(b), the Commissioner is requested to delete the name(s) of the following person or persons who are not inventors of the invention being claimed in this application:
8. ☒ Amend the specification by inserting before the first line the sentence: "This application is a ☐ continuation ☒ division of application number 08/813055, filed 03/07/1997, (status, abandoned, pending, etc.)."

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(REQUEST FOR FILING A PATENT APPLICATION UNDER 37 CFR 1.60, PAGE 2)

9. [X] New formal drawings are enclosed.

10. [ ] Priority of foreign application number _____, filed on _____ in _____ is claimed under 35 U.S.C. 119(a) - (d).
    [ ] The certified copy has been filed in prior application number ___ / _____, filed _____.

11. [ ] A preliminary amendment is enclosed.

12. [X] The prior application is assigned of record to KT Corporation.

13. [ ] Also enclosed:

14. [X] The power of attorney in the prior application is to: Garold E. Bramblett; James R. Frederick; Robert H. Ware; Alfred A. Fressola; and K. Bradford Adolphson.
    a. [X] The power of attorney appears in the original papers in the prior application.
    b. [ ] Since the power does not appear in the original papers, a copy of the power in the prior application is enclosed.
    c. [X] Address all future correspondence to: (May only be completed by applicant, or attorney or agent of record.)

[ ] Customer Number [ ] → *Type Customer Number here*
OR
Place Customer Number Bar Code Label here

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7/22/97
Date

Signature

Garold E. Bramblett
Typed or printed name

[ ] Inventor(s)
[ ] Assignee of complete interest. Certification under 37 CFR 3.73(b) is enclosed.
[X] Attorney or agent of record
[X] Filed under 37 CFR 1.34(a)
Registration number if acting under 37 CFR 1.34(a). 19,119

861-001-009

CONTINUOUS SOLID STATE WEB COATING PROCESS AND WEBS PRODUCED THEREBY

Technical Field

This invention relates to a novel method for the continuous production of a web coated with a layer of a powdered active substance. The active substance is caused to adhere to the web by means of a thermoplastic binder present in a sufficiently small volume that it does not interfere with the adsorbent or otherwise desirable characteristics of the active material.

Background Art

The closest known processes to that of this invention are described in Koslow U.S. Patents No. 5,019,311; 5,147,722; 5,189,092; 5,249,948; and 5,331,037, their parent applications, their corresponding foreign patent applications and patents, and the references cited therein.

The above-mentioned patents disclose processes for the production of composite materials which are characterized by primary particles interconnected by a binder material. Some of these processes require high pressure and shear or extrusion through a die with carefully controlled back pressure. These prior art processes are extremely useful in producing a wide variety of articles including extruded solid forms such as activated carbon filters.

It would often be desirable to impregnate, cover, or otherwise treat a relatively fragile web base material with an active component such as a powdered adsorbent or absorbent material. One example would be a nonwoven medium coated with agents having water absorption and odor adsorption characteristics as in a diaper or hygiene product. A number of other related products will be apparent to those skilled in the art such as, for example, coated paper tissues and toweling, and fabrics such as surgical bandages and sanitary napkins. However,

the fragile nature of the underlying base material would make it impractical to employ the known prior art techniques which require high pressure and shear.

In the prior art referred to above, the powdered active material is formed into a self-supporting sy fusion of a thermoplastic material with which it is intimately mixed. However, the pressures, temperatures, and shear involved, or the process equipment used would not permit their application to fragile substrates such as the webs described herein. Accordingly, it is a primary object of the present invention to provide a method for continuously coating a relatively fragile web with a dry mixture of at least one particulate active material and a very finely divided particulate thermoplastic binder. Other objects, features, and advantages will become apparent from the following description and appended claims.

Disclosure of Invention

In accordance with the present invention a loose, dry composite powder is formed which comprises at least one group of particles of an active ingredient and particles of a thermoplastic binder. The binder particles are quite small in size, preferably on the order of 20 microns and no greater than approximately 40 microns on average. The particle size of the active ingredient may be much larger, within the range, for example, of 5-5000 microns. The small size of the thermoplastic binder particles causes them to adhere to the particles of the active ingredient by electrostatic and van der Waal forces. In addition to their tendency to stick to the active particles, the binder particles also have a high innate cohesion.

The mixture of active and binder powders is applied to the surface of a moving web by means of a knurled roller. The coated web, which can be preheated through a convective or infra-red oven, is then passed through the nip of a pair of rollers, one of which is heated, which apply both heat and pressure to fuse the thermoplastic binder to the active particles and to the underlying web. This step may also be employed to incorporate a second web to achieve a sandwich effect with the active material incorporated between two web surfaces. Upon leaving the heated rollers, the thermoplastic binder sets to form a single, composite structure.

Brief Description of Drawings

FIG. 1 is a schematic diagram illustrating an apparatus for the practice of the method of this invention; and

FIG. 2 is a graph showing the typical particle size distribution of a binder usable in this invention.

Best Mode for Carrying Out the Invention

As has been described above, any of a large number of active particulate agents may be applied to an underlying web in accordance with this invention. Essentially the only limitation relates to the activity desired, e.g. liquid absorption, odor adsorption, medicament delivery, etc. The critical features of this invention, however, reside in the thermoplastic binder which is employed to coalesce the active particles and adhere them to the underlying web. For this purpose, the thermoplastic binder must be in the form of very small particles and must be present in a small enough volume that they do not interfere with the functioning of the active agent. Preferably, the binder will have an effective diameter of not more than 40 microns on average with an optimum size of 20 microns on average. A binder which is suitable for the process of this invention may be produced from normally solid, synthetic organic polymeric thermoplastic resins by the method disclosed in U.S. Patent 3,432,483 of Peoples, et al. Examples of suitable binders are Microthene® F, microfine polyolefin powders produced by Quantum Chemical Company, such as, for example, their low density polyethylene designated FN-510 and their ethylene-vinyl acetate copolymer designated FE-532. Fig. 2 illustrates the typical particle size distribution of Microthene FN-510 powder.

Fig. 1 illustrates an exemplary apparatus for the practice of this invention. A supply roll 10 provides a web 12 of the substrate to be treated, such as a nonwoven tissue or towelling paper. Downstream from supply roll 10 is a knurled roller 13 positioned to receive the composite powder 14 of this invention from a hopper 16 and apply the powder to the upper surface of the web 12. The surface of the knurled roller 13 may be designed to provide a

substantially continuous coating or, alternatively, a coating of a specific design such as, for example, stripes on the web surface. A brush 18 may be employed to aid in removing the composite powder from the knurled roller 13. Thereafter, the web 12 is passed through the nip 20 between a heated idler roller 22 and a drive roller 24. A pneumatic cylinder 26 is connected via a rod 28 to the axle of the idler roller 22 to maintain a desired pressure on the web within the nip 20. In passing over the surface of the heated roller 22, the binder is heated to a temperature equal to or greater than its Vicat softening temperature as it enters the nip 20. Within this nip the binder material fuses under pressure with the active material and with the material of the web. In the illustrated apparatus there is provided a second supply roll 30 of a web 32 which may be of the same or a different material from that of base web 12. This web is also passed between the nip 20 of the rollers 22, 24 and on the top of the particulate material which is being fused. Accordingly, the web 34 which leaves the roller 24 is a composite with both a top and bottom sheet, film, or nonwoven layer. Upon leaving the nip 20, the binder cools and hardens, thereby forming the desired composite. The composite web 34 passes onto a takeup roll 36. Some specific examples of the process of this invention are as follows.

Note: The Vicat softening temperature is defined by Quantum Chemical Company, Cincinnati, Ohio, as ". . . the temperature at which the finished [thermoplastic] article becomes too soft to withstand stresses and keep its shape. It is the temperature at which a flat-ended needle of 1 mm cross section under a load of 1 kg penetrates 1 mm into a . . . specimen. In the Vicat test, the temperature of the specimen is increased at a uniform rate."

Example 1. Iodine Paper.

Iodine paper has utility when used, for example, in a filter unit as a germicidal element.

Both the substrate and the upper layer were 23 cm wide webs of 0.8 oz./sq. yd.spun bonded polyester identified as Reemay type 2016. The production apparatus is as generally shown in Fig. 1 and described above.

The powder mixture consisted of 10% by weight ethylene-vinyl acetate copolymer, (FE532 of Quantum Chemical Company, Cincinnati, OH) and 90% by weight iodinated ion

exchange resin, 47.5% iodine, balance inert, approximately 20-50 mesh particle size (Grade A605 Puradine™ iodinated resin from The Purolite Company, Bala Cynwyd, PA).

The webs moved at the rate of 0.6 m/min and the composite powder was laid down in the amount of .02-.07 g/cm^2. The heated roller was 10 inches in diameter and heated by hot oil to a temperature of 135°C. The binder reached its Vicat softening temperature of 75-80°C in the nip. Pressure in the nip was maintained at approximately 70 kg/cm. The product was a composite medium of good strength and porosity containing nearly 85% by weight of iodated resin. The fact that the resin is not dry prior to processing did not have a significant impact on the quality of the product.

Example 2: Carbon/Soda Paper.

Carbon and sodium-bicarbonate impregnated paper has particular utility as an odor removing component in, for example, an odor adsorbing sheet used in air filtration applications.

The apparatus was substantially identical to that of Example 1. However, the composite powder comprised 17% FE-532. The remaining 83% was 50% 80-325 mesh (500-44μ) activated carbon and 50% 30-40μ particles of sodium bicarbonate ($NaHCO_3$). The web was run at a speed of 0.6-0.9 m/min and powder was deposited at the rate of .015 g/cm^2. The heated roller was at a temperature of 138°C. Three impregnated papers having the same widths as in Example 1 were successfully obtained with (i) both the upper and lower substrates consisting of cellulosic tissue, (ii) both the upper and lower substrates consisting of cellulosic towel stock, and (iii) the lower substrate consisting of cellulosic towel stock and the upper substrate layer consisting of cellulosic tissue stock.

Example 3. Carbon Air or Liquid Filter Paper.

This adsorbent medium has utility in any situation where carbon treatment of either air or liquid is desirable.

The apparatus was similar to that of Example 1. The lower and upper substrates were both spun bonded polypropylene, (Typar grade 135 of Reemay Corporation). The powder mixture was 30% by weight FE-532 and 70% coconut carbon of 80-325 mesh (500-44μ). The heated drum was at a temperature of 150°C and the web speed was 0.6-1.0 m/min. The composite powder was deposited in the amount of .015g/cm^2 . This adsorbent medium was suitable for air filtration. The process was repeated substituting a bituminous coal based carbon for the coconut carbon. The resulting composite medium was optimal for water filtration applications. Both materials were entirely stable when operated in water and did not release fines.

Example 4. Manganese Oxide Paper.

This paper has utility as a filter for removal of heavy metals, such as lead.

The apparatus was substantially identical to that of the preceding examples. Both the lower substrate and the upper layer comprised 25 cm wide Castle® facing spun bonded polypropylene from Kimberly-Clark Corporation. The powder mixture was 17% FE-532 and 83% MnO_2 of average particle size approximately 44μ. Web speed was 0.8-1.5 m/min. Powder lay-down was .015 g/cm^2 and the heated drum temperature was 135°C. The resulting composite medium retains the manganese dioxide in its fully active state where it is capable of oxidizing and precipitating lead, cadmium and other heavy metals.

Example 5. Super-Absorbent Composite.

This product has utility in absorbing liquids and might be used, for example, in diapers.

The apparatus was similar to those described in the preceding examples. Both the lower substrate and the upper layer comprised spun bonded polypropylene from Kimberly-Clark Corporation. The powder mixture was 10% FE-532 and 90% FavorSorb® 880 (a super absorbent acrylic-based polymer obtained from Stockhausen Corporation, Greensboro, NC. Two runs were made as follows, with production of suitable, super-absorbent composites:

(a) The composite powder laydown was .015 g/cm^2. Web speed was 0.8 m/min, the temperature of the heated drum was 138°C, and pressure was approximately 100 psi.

(b) The composite powder laydown was .36 g/cm^2. Web speed was 0.5-0.6 m/min, the temperature of the heated drum was 177°C, and pressure was approximately 100 psi.

This produced a composite medium having excellent water absorption characteristics.

It is believed that the many advantages of this invention will now be apparent to those skilled in the art. It will also be apparent that a number of variations and modifications may be made therein without departing from its spirit and scope. Accordingly, the foregoing description is to be construed as illustrative only, rather than limiting. This invention is limited only by the scope of the following claims.

CLAIMS

1. A process for fusing a particulate active agent to the surface of a substrate web which comprises:

preparing a mixture of at least one particulate active agent with a particulate binder material having an average particle size not exceeding approximately 40 microns;

applying the mixture to the surface of said substrate to produce a uniform or textured surface of powder covering the substrate web;

heating the powder and substrate to at least the Vicat softening temperature of said binder material but below the melting temperature of the substrate and said active agent ingredients; and

thereafter applying pressure to said coated substrate surface to cause the softened binder material to fuse said particulate active agent particles to each other and to said web substrate surface.

2. The process of claim 1 comprising the additional step of depositing upon said mixture an upper layer of sheet material, whereby said substrate web, powder mixture, and upper layer are simultaneously subjected to said application of pressure.

3. The process of claim 1 wherein said pressure is applied by passing the coated web through the nip of a pair of pressure rollers.

4. The process of claim 2 wherein said pressure is applied by passing the coated web and upper layer through the nip of a pair of pressure rollers.

5. The process of claim 1 wherein said binder material is a synthetic organic polymeric thermoplastic resin.

6. The process of claim 5 wherein said binder material is ethylene-vinyl acetate copolymer.

7. The process of claim 5 wherein said resin is polyethylene.

8. The process of claim 7 wherein said resin is low density polyethylene.

9. The process of claim 7 wherein said resin is high density polyethylene.

10. The process of claim 1 wherein said active agent is carbon.

11. The process of claim 1 wherein said active agent is sodium bicarbonate.

12. The process of claim 11 wherrein said active agent comprises activated carbon.

13. The process of claim 1 wherein said active agent is iodated resin.

14. The process of claim 1 wherein said active agent is manganese dioxide.

15. The process of claim 1 wherein said active agent is a liquid absorbent.

16. A first substrate web having a first surface upon which is deposited a particulate iodinated resin and particles of a thermoplastic binder fused to both of said particulate resin and said first surface.

17. The web of claim 16 comprising, in addition, a second substrate web having a second surface spaced from said first substrate web and fused to said thermoplastic binder.

18. A first substrate web having a first surface upon which is deposited particulate carbon and particles of a thermoplastic binder fused to both of said particulate carbon and said first surface.

19. The web of claim 18 comprising, in addition, a second substrate web having a second surface spaced from said first surface and fused to said thermoplastic binder.

20. A first substrate web having a first surface upon which is deposited particulate sodium bicarbonate and particles of a thermoplastic binder fused to both of said particulate sodium bicarbonate and said first surface.

21. The web of claim 20 comprising, in addition, a second substrate web having a second surface spaced from said first surface and fused to said thermoplastic binder.

22. A first substrate web having a first surface upon which is deposited particulate manganese oxide and particles of a thermoplastic binder fused to both of said particulate manganese oxide and said first surface.

23. The web of claim 22 comprising, in addition, a second substrate web having a second surface spaced from said first surface and fused to said thermoplastic binder.

24. A first substrate web having a first surface upon which is deposited a particulate liquid absorbent and particles of a thermoplastic binder fused to both of said particulate liquid absorbent and said first surface.

25. The web of claim 24 comprising, in addition, a second substrate web having a second surface spaced from said first surface and fused to said thermoplastic binder.

1265.052

CONTINUOUS SOLID STATE WEB COATING PROCESS AND WEBS PRODUCED THEREBY

ABSTRACT

One or more particulate active agents are fused to the surface of a substrate web by mixing the particulate agents with a particulate binder having a particle size not exceeding an average diameter of approximately 40 microns and coating the composite mixture onto the surface of the substrate. Thereafter, the coated substrate is heated to a temperature equal to or greater than the Vicat softening temperature of the binder and compressed within the nip of a pair of pressure rolls to achieve fusion. If desired, a top layer may be placed upon the coated composite prior to the compression step. Also disclosed are various products manufactured by the process.

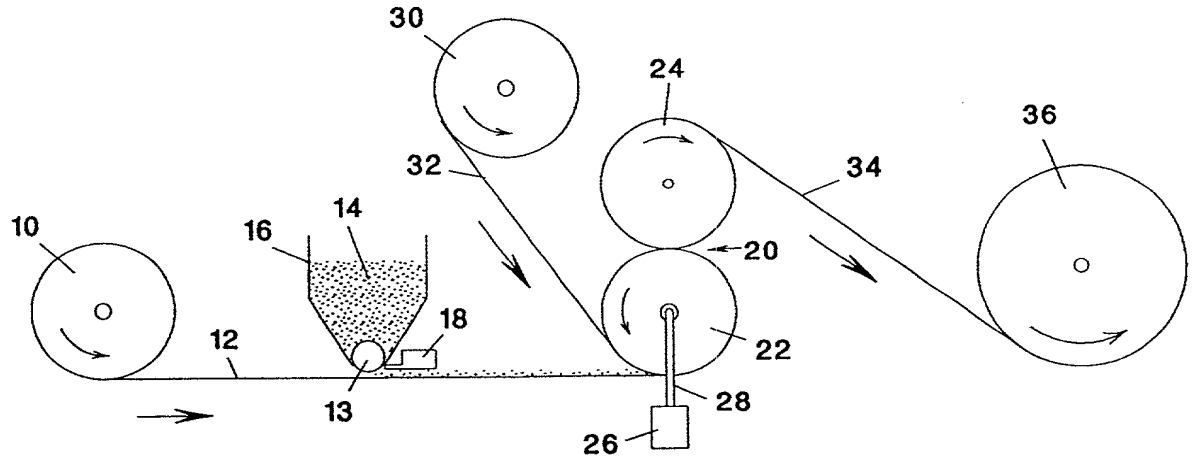
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FIG. 1

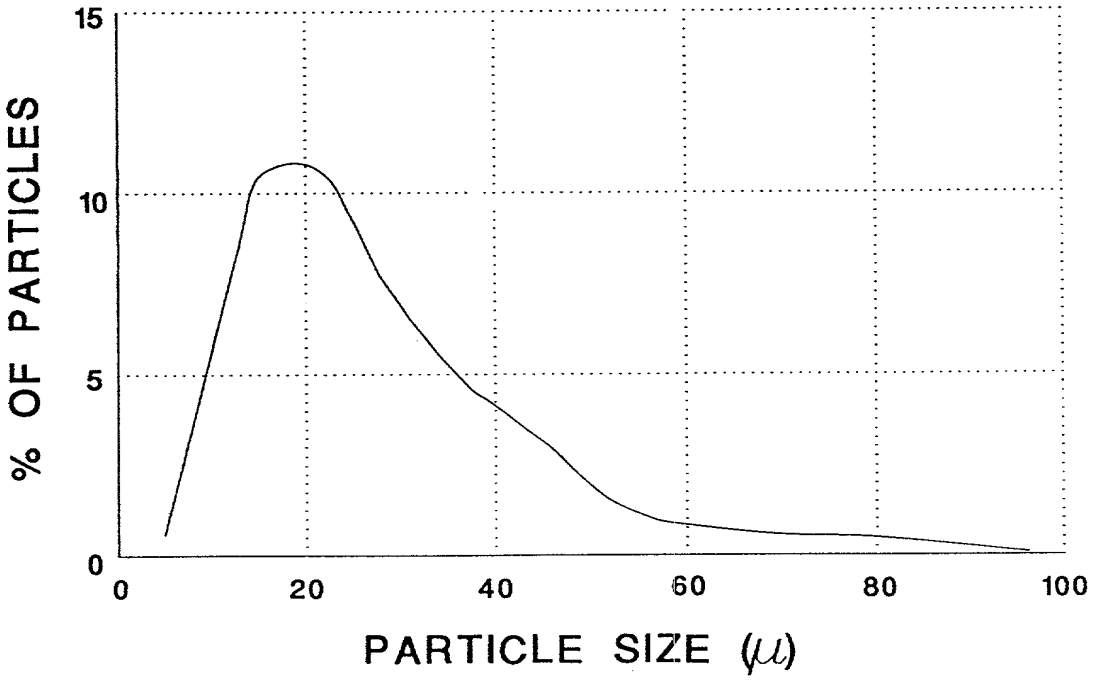
15
10
5
0
% OF PARTICLES
0
20
40
60
80
100
PARTICLE SIZE (μ)

FIG. 2

DECLARATION FOR PATENT APPLICATION Docket No. [illegible]

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled CONTINUOUS SOLID STATE WEB COATING PROCESS AND WEBS PRODUCED THEREBY, the specification of which

(check one) ☒ is attached hereto.

☐ was filed on _____ as Application Serial No. _____ and was amended on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s)			Priority Claimed	
(Number)	(Country)	(Day/Month/Year Filed)	Yes	No
(Number)	(Country)	(Day/Month/Year Filed)	Yes	No
(Number)	(Country)	(Day/Month/Year Filed)	Yes	No

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.)	(Filing Date)	(Status—patented, pending, abandoned)
(Application Serial No.)	(Filing Date)	(Status—patented, pending, abandoned)

I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith with full power of substitution and revocation: Garold E. Bramblett Reg. No. 19,119; James R. Frederick Reg. No. 25,865; Robert H. Ware, Reg. No. 18,179; Alfred A. Fressola, Reg. No. 27,550; and K. Bradford Adolphson, Reg. No. 30,927

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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Inventor's signature: Gordon Spilkin Date: 3/7/97
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Post Office Address: ~~19 Woodway Rd.~~, Stamford, CT 06907
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Full name of _____ inventor _____
Inventor's signature _____ Date _____
Residence _____ Citizenship _____
Post Office Address _____

Full name of _____ inventor _____
Inventor's signature _____ Date _____
Residence _____ Citizenship _____
Post Office Address _____